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Dilatometric determination of thermal diffusivity in low conducting materials

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Abstract. Thermal diffusivity of low conducting materials is measured by detecting the thermal expansion of a cylindrical specimen heated at one base, in contact with a copper disc. The simultaneous recording of the temperature of the copper and of the capacitive signal by which we measure the dilation of the specimen allows the experimental determination of thermal diffusivity without the uncontrolled heat losses which usually occur due to temperature sensors.

1. Introduction

Measurements of thermal diffusivity in low conducting materials are difficult because any uncontrolled heat loss or heat source, which would be small for experiments involving metallic specimens, give rise – in this case – to thermal fields having a magnitude comparable to that of the thermal field to be detected.

Among the sources of error one has to consider are the heat lost or gained by (i), radiation through the surface of the sample; (ii), conduction through the mechanical support of the specimen; (iii), conduction through the thermocouple leads.

The new method proposed in this paper consists of deducing the thermal diffusivity by measuring the thermal expansion of a vertical cylindrical sample heated at the lower base. We use a high-sensitivity capacitive system to measure the thermal dilation of the sample: using this system it is possible to measure displacements of the order of 0.1 nm. In more precise terms, the sample, of height l, is put on a copper disc, D_c lying on a large base of fused silica, B (see figure 1). The sample P_1 is surrounded by a thermal guard, represented by a hollow cylinder of the same material, P_2, having the same external diameter as the copper disc, the radial gap between the two cylinders being as small as possible (in our experiments it was about 0.5×10^{-3} m). The copper disc is surrounded by an insulated electrical wire J, which can be used as a heat source by switching on the current generator to which it is connected. A thermocouple inserted into the copper disc allows the recording of its temperature as a function of time. On the upper base of the central specimen a fused silica tripod (with three pins at 120°C) supports a fused silica disc D_1, having the whole surface coated by a conducting film of tin oxide. It forms a capacitor with a hollow fused silica disc D_2, which is similarly coated on its lateral border and its upper surface: the distance between D_1 and D_2 can be adjusted by means of three levelling screws T pressing on a hollow cylinder of fused silica, H, supporting D_2 and fixed to basement B.

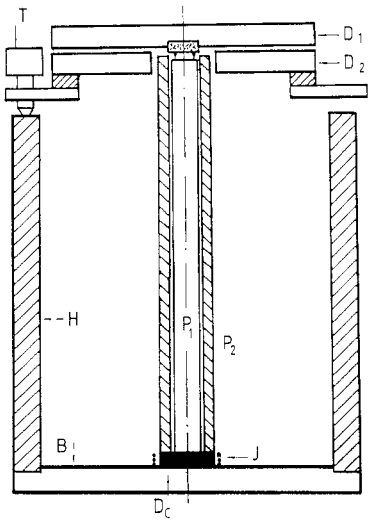

Figure 1. Schematic view of the experimental apparatus.

The capacitive signal may be taken directly from the conducting borders of the two discs D_1 and D_2: in this case care has to be taken to avoid spurious momenta applied by the electric lead to the disc D_1, which is in critical equilibrium on the sample. In our measurements at room temperature we obtained a satisfactory contact by attaching a piece of nickel wire, having the form of a Γ, to the border of D_1 with the vertical leg immersed in mercury. Another nickel wire, immersed into the same vessel containing mercury, was connected to the BNC cable leading to the electronic measuring apparatus. In this way, the electrical connection was obtained through the liquid, without any mechanical perturbation of the

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capacitor plates. In temperature ranges in which the use of mercury is not applicable, the connection could be easily obtained by a short piece of vertical wire hanging from a fixed support in the proximity of the centre of the upper face of D_1. The capacitive signal is measured by means of an apparatus with high sensitivity (10^{-6}) (Barbero and Strigazzi 1983): it is brought through a GenRad impedance bridge to an Ithaco lock-in amplifier and finally to a $Y(t)$ recorder, which also receives the amplified signal from the copper-disc thermocouple. The whole apparatus is put on the basement of a vacuum box, the upper part of which can be raised or lowered through a hydraulic pump: all the electrical connections are obviously inserted into a fixed part of the box.

The simultaneous recording of the temperature of the copper disc (θ^{Cu}) and of the capacitive signal (V) related to the thermal expansion of the sample allows, as we will show, the experimental determination of its thermal diffusivity α. The analysis required to infer α from $\theta^{Cu}(t)$ and $V(t)$ is given in the following section. Here we wish only to stress the advantages of our method with respect to the other methods described in the literature:

(i) because the thermocouple is inserted into the copper disc, any heat conveyed by the thermocouple leads is immediately spread through the whole disc, owing to the high thermal conductivity of copper; thus, there is no doubt that the instantaneous temperature measured by the thermocouple is the temperature of the whole copper disc. This circumstance provides a well defined boundary condition for our problem: the whole base of the sample is in contact with a system whose temperature is uniform and unambiguously known as a function of time.

(ii) The hollow sample P_2 acts as a thermal guard for the sample P_1 under study, and eliminates any heat exchange through the lateral surface of P_1. This is because the inner part of P_2 and the outer part of P_1 are heated in the same way and, consequently, the radiation heat lost by P_1 is balanced by the heat received from P_2.

(iii) The mechanical support of the sample being represented by the copper disc means that any uncontrolled perturbation produced by the support itself is automatically eliminated.

(iv) Since the material under study has a low conductivity, it is possible to make measurements in a time interval shorter than $l^2/\pi^2\alpha$, which represents approximately the time required by the heat front to go from $z = 0$ to $z = l$ (Danielson and Sidles 1969). Under these conditions the heat lost through the upper base of the sample is rigorously zero. If the material is a good thermal conductor, the heat front arrives at $z = l$ before the end of the measurement, but also in this case the radiation heat loss is negligible below room temperature. In fact, the dimensionless parameter of interest for the heat loss is hl/k, where h is the exchange coefficient of Newton's law (containing the emissivity) and k the conductivity: for metallic samples, k is so high that radiation heat becomes important only at high temperatures. The use of the method for metallic samples, though not strictly necessary, may derive from calibration procedures which will be discussed in § 3. In this case, however, it is necessary to avoid heat conduction through the contact between the sample and the upper disc D_1. The fused silica tripod satisfies this requirement because the thermal diffusivity of fused silica is low and, in any case, the contact area between a fused silica pin and the sample is essentially a point, i.e. zero.

(v) Since we use the dilation of the sample as a thermal field detector, we have no sensor producing uncontrolled perturbations to the heat flow. Essentially, the thermal field in a given region of the sample is measured by a device (the capacitor) which is put at a great distance, i.e. at a place where it does not interfere with the field. Such a peculiarity cannot be achieved with the usual thermal detectors such as thermocouples.

Summarising, we present a method in which the boundary conditions are perfectly known and the uncontrolled heat losses are eliminated: it has to be considered a step forward with respect to the method already used in our laboratory for measurements of thermal diffusivity in metals (Omini *et al* 1989). Care has only to be taken to avoid confusion between the measured temperature of the copper disc (θ^{Cu}) and the specimen temperature (θ_b) at the base in contact with it. The interface layer gives rise to a temperature drop which is generally assumed to be proportional to the instantaneous heat flow (U) through the layer, namely (Moses and Johnson 1988, Beck 1988)

$$\theta^{Cu} - \theta_b = U/H \tag{1}$$

where H depends on the nature of the contact. We will take into account this condition in solving the heat diffusion equation.

A final comment concerns the possibility of supplying power to the lower part of the sample without using the copper disc and the thermocouple. To this purpose we stress the following points:

(i) in principle, the dilatometric method is usable for any experimental arrangement in which the temperature of the specimen at the lower base is measured by a sensor which simultaneously acts as a heater. Although a detailed description of a system of this kind may represent part of future research, we think that methods involving silver film depositions on the base may be of interest, because they can be used not only to generate heat, but also to deduce the temperature of the adjacent layer from a measure of the electrical resistance of silver (Cahill and Pohl 1987, Gustafsson and Karawacki 1983).

(ii) The thermocouple can be spread over the whole base of the sample by means of two adjacent films (for instance, of copper and constantan, see Théry and Maréchal 1980) This device would strongly reduce radial temperature gradients due to unwanted heat conveyed by the thermocouple leads. In this case, the heater can be represented by a resistive film electrically insulated from the thermocouple by an interposed non-conducting film.

(iii) Alternatively, the method can be employed for a specimen heated by a laser pulse impinging on its lower base. In this case the heat source may be regarded as a

known function of time, exactly as in the flash method (see Danielson and Sidles 1969). Consequently, since a knowledge of the temperature at the base is not required, the thermocouple is eliminated from the experimental apparatus, and the measured time dependency of the capacitive signal alone is sufficient to determine the diffusivity. In spite of this attractive possibility, however, we point out that, in order to allow the dilatometric measurement, the support of the specimen must be somehow connected with the lower base, in which case it not only exchanges unknown amounts of heat with the specimen, but also unavoidably interferes with the laser beam, thus destroying the uniformity of the beam distribution on the base.

The length of time required for sample preparation by the methods considered in (i) and (ii) and, on the other hand, the difficulties connected with the specimen support as discussed in (iii) lead the authors to believe that the experimental arrangement presented in figure 1 is probably the simplest at our disposal for practical applications.

2. Theory

Since the base of the specimen is uniformly heated and, on the other hand, heat exchanges through the lateral surface are not allowed, the problem is one dimensional. Let us introduce a coordinate system with the origin at the centre of the base in contact with the copper disc and the z axis directed towards the upper base ($z = l$). The solution of the Fourier equation referring to a *constant* heating rate through the lower base, $S = -k(\partial\theta/\partial z)_{z=0}$, is given in standard texts, e.g. Carslaw and Jaeger (1959). To extend the solution to a general heating rate, the whole time interval of an experimental heating curve can be subdivided into many small intervals of equal width, each of them being characterised by a constant value of S, depending, however, on the time interval itself. Let us indicate by S_m the heating rate referring to the interval between t_m and t_{m+1} and by $\theta_m^{m+1}(z, t)$ the corresponding temperature field: one has

$$\theta_m^{m+1}(z, t) = (\alpha s_m/kl)t + (S_m/2kl)(z-l)^2 + \sum_n c_n^m \cos(n\pi z/l)\exp(-\alpha n^2\pi^2 t/l^2) \tag{2}$$

where k is the thermal conductivity, α the diffusivity and m and n are integers: equation (2) accounts implicitly for the condition $-k(\partial\theta/\partial z)_{z=l} = 0$ at the upper base.

Imposing the conditions $\theta_0^1(z, 0) = 0$ and $\theta_{m-1}^m(z, t_m) = \theta_m^{m+1}(z, t_m)$ $(m \geqslant 1)$ and putting $\theta_m^{m+1}(z, t_{m+1}) = \theta_{m+1}(z)$ one finds, after a standard analysis,

$$\theta_{m+1}(z) = (S_m/kl)[\alpha t_{m+1} + \tfrac{1}{2}(z-l)^2] - (S_0 l/2k)G_z(t_{m+1}) - (l/k)\sum_{p=1}^{m}(S_p - S_{p-1}) \times [\alpha t_p/l^2 + \tfrac{1}{2}G_z(t_{m+1} - t_p)] \tag{3}$$

for $m \geqslant 1$ and

$$\theta_1(z) = (S_0/kl)[\alpha t_1 + \tfrac{1}{2}(z-l)^2] - (S_0 l/2k)G_z(t_1) \tag{4}$$

where

$$G_z(t) = (4/\pi^2)\sum_{n=1}(1/n^2)\cos(n\pi z/l)\exp(-\alpha n^2\pi^2 t/l^2) + \tfrac{1}{3}. \tag{5}$$

Now if θ_{m+1}^{Cu} is the temperature of the copper disc at t_{m+1}, condition (1), owing to equations (3) and (4), becomes

$$\theta_{m+1}^{Cu} - (\theta_{m+1})_{z=0} = (1/H)S_m. \tag{6}$$

Putting $lS_m/k = Q_m$, $k/Hl = \gamma$, one deduces from equations (3)–(6) that

$$\theta_{m+1}^{Cu} = (\gamma + \tfrac{1}{2} + \alpha t_{m+1}/l^2)Q_m - \tfrac{1}{2}Q_0 G_0(t_{m+1}) - \sum_{p=1}^{m}(Q_p - Q_{p-1})[\alpha t_p/l^2 + \tfrac{1}{2}G_0(t_{m+1} - t_p)] \tag{7}$$

for $m \geqslant 1$ and

$$\theta_1^{Cu} = [\gamma + \tfrac{1}{2} + \alpha t_1/l^2 - \tfrac{1}{2}G_0(t_1)]Q_0. \tag{8}$$

For a one-dimensional temperature distribution the thermal dilation of the sample, as determined through a rigorous solution of the thermoelastic equations (Kovalenko 1969), is found to be indistinguishable, within a few parts in 10^3, from that predicted by the simple expression

$$\beta\int_0^l \theta(z, t)\,dz \tag{9}$$

where β is the linear thermal expansion coefficient. Summing the contribution due to the copper disc we obtain the total change in the gap between the two capacitor plates at t_{m+1}

$$\Delta_{m+1} = \beta\int_0^l \theta_{m+1}(z)\,dz + \beta_{Cu} b\theta_{m+1}^{Cu} \tag{10}$$

where $b = 6 \times 10^{-3}$ m is the thickness of the copper disc and $\beta_{Cu} = 1.68 \times 10^{-5}\ K^{-1}$ its thermal expansion coefficient. Introducing (3) and (4) into (10) gives

$$\Delta_i = \beta l F_i + \beta_{Cu} b\theta_i^{Cu} \tag{11}$$

where

$$F_1 = Q_0\alpha t_1/l^2 \tag{12}$$

$$F_i = (\alpha t_i/l^2 + \tfrac{1}{6})Q_{i-1} - Q_0/6 - \sum_{p=1}^{i-1}(Q_p - Q_{p-1})(\alpha t_p/l^2 + \tfrac{1}{6}) \tag{13}$$

with $i \geqslant 2$.

At this point the procedure to be followed consists, as a first step, of making an arbitrary choice for the unknown parameters α and γ and solving systems (7) and (8) with respect to the heat sources Q_i. A computer can be easily programmed for this operation, because one immediately obtains Q_0 from (8) and $Q_1, Q_2, \ldots$ from (7) written for $m = 1, m = 2, \ldots$, respectively. As a second step one must introduce the heat sources into (11)–(13) in order to obtain the theoretical time dependency of the change of gap, Δ_i. Let us now impose the best fit of Δ_i to the

experimental change of gap $(\Delta_i)_{exp}$: this amounts to minimising the expression

$$\delta = \sum_{i=1}^{N} (\Delta_i - (\Delta_i)_{exp})^2 \qquad (14)$$

where the sum is extended to the N times t_i taken at regular intervals through the whole time interval for which the simultaneous experimental determination of $\theta^{Cu}(t)$ and $\Delta_{exp}(t)$ has been made. By exploring the behaviour of δ in the plane (α, γ) we can determine numerically the extreme values of the above parameters for which δ reaches its absolute minimum.

3. Calibration of the instrument

We assume $\Delta_{exp}(t)$ to be linked to the capacitive signal $V(t)$ by a non-linear relation of the form

$$\Delta_{exp} = \rho V + \sigma V^2 \qquad (15)$$

where ρ and σ can, in principle, be obtained through optical methods: in fact, the upper face of disc D_1 could be used as the mobile mirror of a Michelson interferometer and the numbers of displaced fringes at two different times t_1 and t_2 compared with the corresponding signals $V(t_1)$ and $V(t_2)$ to deduce the two parameters. An alternative method, which was followed for the measurements described in the present paper, is based on the fact that ρ and σ do not depend on the physical properties of the sample, but only on the geometry of the capacitor plates, that is, if the plates are parallel, on the initial gap between them: since the gap is strictly related to the capacitance, we are, in principle, concerned with the same couple of values (ρ, σ) in two independent experiments where the samples are different, but the levelling screws T have been adjusted so as to obtain the same initial value of capacitance, C_0. This feature allows a simple determination of ρ and σ, by studying the heat propagation in a reference sample with known values of diffusivity and thermal expansion coefficient: in this case the analysis of the heating curves $\theta^{Cu}(t)$ and $V(t)$ immediately provides ρ and σ (see § 4).

For such a method of calibration it is essential to ensure a good parallelism between the two capacitor plates: only in this way, in fact, can two different mountings of the apparatus, referring to the same value of capacitance, be referred to the same couple of values of ρ and σ. Experiments performed with the same specimen of Al on different mountings of the apparatus showed that the two capacitor plates are in satisfactory parallelism if the following criterion is adopted: an equal number of turns produced independently on each levelling screw (sensitivity 5×10^{-7} m) produces the same capacitive signal within 10%. In fact, working with geometrical configurations satisfying this condition, we found that, for the same specimen and for the same initial value of capacitance, the values of ρ were reproducible within 2% and the values of σ within 20% (though rough, such a determination of σ is sufficient for our purposes, since the non-linear term is very small).

4. Experimental procedure and results

To calibrate the instrument, we first determined ρ and σ by subjecting a sample of pure aluminium ($R = 0.5 \times 10^{-2}$ m, $l = 15 \times 10^{-2}$ m) to the heating experiment described by the continuous curves of figure 2 corresponding to $C_0 = 382$ pF and $T = 20$ °C. These were obtained by switching the heater on and off three times in succession.

Since, for aluminium, α is known, one can use the information contained in the two curves to obtain ρ and σ. To this purpose, it is convenient to put $\lambda = \rho/\beta l$, $\mu = \sigma/\beta l$ and to minimise, instead of (14), the square sum

$$\delta' = \sum_{i=1}^{N} (F_i + (\beta_{Cu}/\beta)(b/l)\theta_i^{Cu} - \lambda V_i - \mu V_i^2)^2 \qquad (16)$$

to be considered in this case as a function of λ, μ and γ. The minimisation with respect to λ and μ can be performed analytically and the remaining minimisation with respect to γ numerically.

We analysed the curves of figure 2 by subdividing the whole time interval of 360 s into 216 parts: we considered this choice sufficient for our purposes, because passing from $N = 216$ to $N = 324$ we did not find any appreciable change in our results. We performed the analysis for two values of the diffusivity of aluminium, namely $\alpha_1 = 0.86 \times 10^{-4}$ and $\alpha_2 = 0.92 \times 10^{-4}$ m^2 s^{-1}: these correspond approximately to the maximum discrepancy between the results of different workers. The extreme values were found to be $\gamma = 13$, $\lambda = 0.924$, $\mu = 6.2 \times 10^{-4}$ for $\alpha = \alpha_1$ and $\gamma = 17$, $\lambda = 0.927$ and $\mu = 6.7 \times 10^{-4}$ for $\alpha = \alpha_2$ (the units of measurement of λ and μ depend on the features of our $Y(t)$ recorder, where we read θ^{Cu} and V, and do not require to be specified in detail). Since the discrepancy between the two couples of values of λ and μ is smaller than the uncertainty (of the order of 2% for λ and 20% for μ) deriving from the degree of reproducibility of the geometrical configuration of the capacitor plates, it turns out that for calibration purposes the actual but approximate known value of the diffusivity of aluminium is widely sufficient. The physical meaning of the numerical value obtained for γ is clearly shown in figure 3, where we plot, for $\alpha = 0.86 \times 10^{-4}$ m^2 s^{-1}, $\gamma = 13$, the time depen-

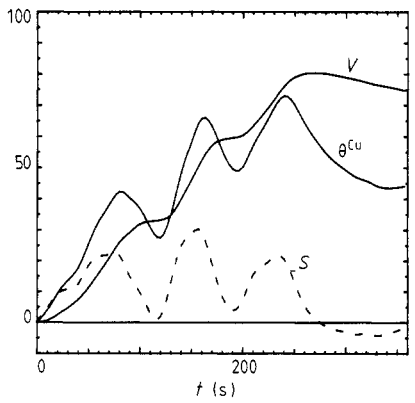

Figure 2. Heating curves obtained for the sample of aluminium. The broken curve is the heat source $Q(t)$ deduced from the solution of equations (7) and (8).

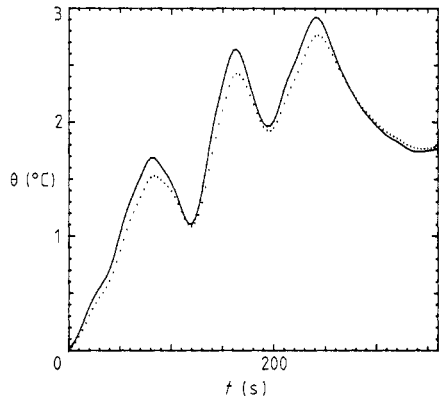

Figure 3. Time behaviour of the temperature of the copper disc (full curve) and of the specimen base (dotted curve) for our experiment on Al.

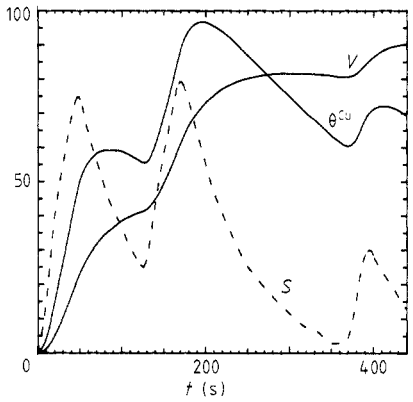

Figure 4. Heating curves obtained for the sample of Plexiglass. The broken curve is the heat source $Q(t)$ deduced from the solution of equations (7) and (8).

dency of the temperature at $z = 0$ in the sample of aluminium, to be compared with the corresponding behaviour of the temperature in the copper disc. The fact that at the end of the time interval, the former exceeds the latter, shows that in this case the copper disc works as a heat sink, because the heater has been switched off and heat has been lost through the underlying base of fused silica, and especially through the heating wire.

A by-product of our method is, therefore, the determination of the temperature jump due to the contact layer at the base of the sample: this is not a trivial result, since the experimental methods presented by the literature on this subject are generally based on the use of many thermocouples which disturb the heat flow in the neighbourhood of the junction (Moses and Johnson 1988). The magnitude of γ strongly depends on the nature of the contact and on the pressure acting on the contact area. In our case we had no pressure, but the two parts were cemented by a conducting glue.

Having calibrated the instrument (that is, determined λ_{Al} and μ_{Al}), we performed a second experiment using a sample of Plexiglass of the same length as the sample of aluminium, We chose Plexiglass because it is a reference material for which recent diffusivity data are available (Kougbeadjo and Théry 1982). The values of λ and μ referring to this new situation are given by

$$\lambda_p = (\beta_{Al}/\beta_p)\lambda_{Al} \qquad \mu_p = (\beta_{Al}/\beta_p)\mu_{Al} \qquad (17)$$

provided that the initial capacitance C_0 is the same in the two experiments on Plexiglass and aluminium. In practice, such a circumstance is easily realised if one has taken care of performing many heating experiments on aluminium at different values of capacitance, so as to have available, by interpolation, the values of λ_{Al} and μ_{Al} corresponding to the experimental value of C_0 in the experiment on Plexiglass. The heating curves corresponding to $C_0 = 382$ pF are reproduced in figure 4. The linear expansion coefficient of Plexiglass at room temperature is $\beta_p = 7.2 \times 10^{-5}\ K^{-1}$ and consequently the ratio β_{Al}/β_p to be used in (17) is 0.343. Inserting into (17) the couple (λ_{Al}, μ_{Al}) corresponding to $\alpha_{Al} = 0.86 \times 10^{-4}\ m^2\ s^{-1}$, one obtains $\lambda_p = 0.308$, $\mu_p = 2.1 \times 10^{-4}$. These values can be used directly in expression (16), which has now to be evaluated as a function of α and γ in reference to the curves of figure 4. The behaviour of δ' in the plane (α, γ) shows a very deep minimum which is, therefore, suitable for determining the diffusivity with remarkable precision. The extreme value of α turns out to be $8.8 \times 10^{-8}\ m^2\ s^{-1}$, in full agreement with the measurements in the literature, giving diffusivity values in the range 7.7×10^{-8}–$1.07 \times 10^{-7}\ m^2\ s^{-1}$. The extreme value of γ, that is *zero* within the experimental uncertainties, is consistent with the fact that γ is, by definition, proportional to the thermal conductivity, which for Plexiglass is very low. This result shows that if the contact between the copper disc and the low conducting sample is established, as in our experiment, with the help of a glue with good thermal conduction, the temperature of the sample at $z = 0$ may be assumed to be the same as the temperature of the copper disc itself.

If the analysis of the curves of figure 4 is repeated by assuming, for λ_p and μ_p, values differing by 2% and 20%, respectively, from the ones used previously, one finds that the extreme value of α is correspondingly altered by about 5%. This is, therefore, the uncertainty that is expected to affect our determination of α, due to the lack of repeatability of the geometry of the capacitor. A different set of measurements, repeated on the same sample of Plexiglass, has fully confirmed this expectation.

A set of measurements was also performed on a sample of Teflon ($\beta = 15 \times 10^{-5}\ K^{-1}$) at 385 pF, a value of capacitance for which we had previously determined the pair of parameters $\lambda_{Al} = 0.897$ and $\mu_{Al} = 5.1 \times 10^{-4}$ for aluminium. The extreme value of the diffusivity was $0.97 \times 10^{-7}\ m^2\ s^{-1}$, in good agreement with the value of $1.1 \times 10^{-7}\ cm^2\ s^{-1}$ given by Shapira and Lichtman (1979). The extreme value of γ was again found to be negligibly small.

5. Conclusion

The method presented in this paper, which can be applied in a wide temperature range from cryogenic to

melting, employs a dilatometric apparatus to determine the thermal diffusivity of low conducting materials. It minimises the uncontrolled heat exchange between the specimen and the environment and is also recommended because only small specimens and a short time interval are required for the measurement. The high sensitivity of the capacitive system, by which we could appreciate displacements of the order of 0.1 nm, was never used in our measurements on Plexiglass and Teflon at room temperature. However, it should be invoked for materials with small thermal expansion coefficients, or, more generally, for low-temperature measurements. An interferometric method of calibration of the capacitor, in which the upper face of disc D_1 is used as the mobile mirror of a Michelson interferometer, is in progress in our laboratory and will be published elsewhere.

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